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# Shear Bond Strength of Self-Etching Adhesive Systems to Er:YAG Laser-Prepared Dentine with and without Pulpal Pressure Simulation

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## Abstract

**Objective:** This study was conducted to study the role of pulpal pressure on the shear bond strength of composite resin bonded to Er:YAG laser-prepared or bur-prepared dentine surfaces using a self-etching adhesive system. **Materials and Methods:** The occlusal surfaces of 80 human third molars were ground flat to expose the dentine. The dentine was prepared using either a carbide bur or an Er:YAG laser at 350 mJ/pulse and 10 Hz (fluence 44.5 J/cm<sup>2</sup>). Clearfil SE Bond was then applied with or without pressure. Rods of composite resin were bonded to dentine surfaces and shear bond tests were carried out. **Results:** When the Clearfil SE Bond was used without pressure, the difference between the shear bond strength values of bur-prepared and laser-prepared dentine surfaces was significant. When the Clearfil SE Bond was used with pressure, the difference of shear bond strength values was not significant between the two types of surface preparation. **Conclusion:** The absence of smear layer formation during the preparation of the dentine by the Er:YAG laser did not improve the adhesion values of self-etching adhesive systems.

## Introduction

ERBIUM:YTTTRIUM-ALUMINUM-GARNET (Er:YAG) laser irradiation was first used 10 years ago as a new method for preparing dental hard tissues. This technique is proposed to replace the high-speed air turbine and low-speed drills. The Er:YAG laser emits energy in the mid-infrared region (2.94  $\mu\text{m}$ ) that falls in an area of the spectrum where dental tissues have absorption peaks (high absorption by water and hydroxyapatite).<sup>1</sup> This laser was used for enamel/dentine surface treatment and removal of carious lesions in an effort to advance minimally-invasive dentistry.<sup>2</sup> To protect the pulp from dangerous overheating, a cooling medium (a mixed water and air spray) is used.<sup>1</sup> The Er:YAG laser is a good way to safely remove hard dental tissues. After preparation with this technique the irradiated dentine acquires microscopically irregular surfaces and open dentinal tubules, without a smear layer.<sup>3</sup> Studies have been performed on the bond strength of composite resin to Er:YAG laser-irradiated dentine.<sup>4</sup> The results have shown that bond strength increases when the surface is acid etched before bonding.

Dentinal moisture, as well as regional structural differences, are important factors that affect the bond strength to dentine.

Thus pulpal pressure may influence the degree of intrinsic wetness of the dentine surface.<sup>5</sup> With new self-etching adhesive systems, the dentinal smear layer is no longer completely eliminated, but is instead treated like a substrate.<sup>6</sup> One-step self-etching adhesive systems, also called “all-in-one” systems, contain an etching agent, a primer, and an adhesive in a single agent. The adhesion values to irradiated dentine surfaces with these new materials are still being analyzed.<sup>7</sup> The adhesive we used in this study (Clearfil SE Bond) is a self-etching adhesive treatment packaged in two bottles: one contains the acid primer and the other contains the adhesive.

The purpose of this study was to measure the bond strength of composite resin to Er:YAG-prepared dentine using self-etching with or without pulpal pressure versus the bond strength to bur-prepared dentine. The presence of resin tags with or without pressure was also assessed to study their influence on shear bond strength.

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## Material and Methods

Eighty freshly extracted caries-free human third molars were used for this *in vitro* study. They were hand scaled, cleaned, and stored in distilled water at 4°C for up to 1 mo. These teeth were then randomly divided into two groups of 40 teeth each. Collection of these samples conformed to a protocol that satisfied the ethical standards set forth by the Centre Hospitalier Universitaire de Nice. The teeth were extracted for periodontal reasons and were obtained from patients who orally consented to their use for research purposes.

In the first group, the apices were cut to 3 mm and the pulp tissue was removed. A cylindrical hole was made in the pulp cavity, and a metal tube was inserted into the hole and glued in place.

The teeth in both groups were embedded in epoxide resin (Epoxycure resin; Buehler Ltd., Lake Bluff, IL, USA). Flat dentine surfaces were obtained by transverse sectioning of the crowns at a distance of 4 mm from the occlusal surface, using a slowly rotating diamond blade under running water (Isomet 2000; Buehler Ltd.).

The dentinal surfaces of 40 teeth in the first group (20 with the metal tube and 20 without; bur-prepared group) were prepared using a tungsten carbide bur (ref. H1.204.014; Komet, Gebr Brasseler, Lemgo, Germany) at high-speed under water cooling. The dentinal surfaces of 40 teeth (20 with the metal tube and 20 without; laser-prepared group) in the second group were prepared using an Er:YAG laser (Key 3™; Kavo Dental, Biberach, Germany), with an output energy of 350 mJ per pulse and a frequency of 10 Hz. The laser beam was delivered perpendicularly to the dentinal surface, with a mirror handpiece (2060™; Kavo Dental) at a distance of 12 mm. This distance was manually maintained in the same manner used in clinical practice. The spot diameter was 0.8 mm (fluence of 44.5 J/cm<sup>2</sup>) and the time of irradiation was 15 sec. Cooling was achieved using an air/water spray. More details about the irradiation parameters can be found in references 8 and 9. After preparation all the teeth were air dried.

The intradental pressure is the pressure on the dentine imposed by the pulpal parenchyma. To simulate this pressure each tooth with a metal tube was connected by a flexible silicone hose to a water bottle placed vertically 35 cm above the sample. The system was evacuated with a vacuum pump and filled with bubble-free water (Fig. 1). The pulpal pressure device we used was based on an original idea proposed by Krejci et al.<sup>10</sup> Pulpal pressure is equivalent to 14 cm of water, and the intradental pressure is equivalent to 34 cm of water.

The same self-etching adhesive system (Clearfil SE Bond™; two bottles, batch #41284; Kuraray, Kurashiki City, Japan) was used for all teeth, and the teeth were prepared according to the manufacturer's recommendations. The self-etching primer was first applied to the dentinal surface. After a 20-sec resting period, the samples were briefly air dried. The adhesive resin was then applied, briefly air dried, and photopolymerized for 10 sec (Astralis 5™; Vivadent, Schaan, Liechtenstein).

A plastic ring (internal diameter of 5 mm) was placed on the dentinal surface of each tooth. A microhybrid composite resin (Spectrum TPH™, batch #0012000850; Detry-Dentsply, Konstanz, Germany) was applied in three 1-mm layers, and each layer was photopolymerized for 40 sec (Astralis 5™, Vivadent), reaching 3 mm in total height. The plastic ring was then removed and excess adhesive was removed using a polishing disc (Sof-Lex™; 3M Espe, St. Paul, MN, USA). The base of each sample was checked using an optical microscope (Olympus VMZ, Tokyo, Japan). The samples were then stored in distilled water at 37°C for 48 h.

The shear bond strength measurements were carried out using a universal tensile testing machine (Erischen, Wuppertal, Germany) provided with a 2-kN sensor, at a velocity of 0.02 mm/sec<sup>-1</sup>. The fractured samples (dentine surfaces and composite resin rods) were placed on aluminum stubs and dried for 7 d in a drying oven at 37°C. They were then gold sputter-coated (JFC-1100E ion sputter; JEOL, Tokyo, Japan). The coated specimens were examined under a scanning electron microscope (SEM) (JSM-5310LV; JEOL) to de-

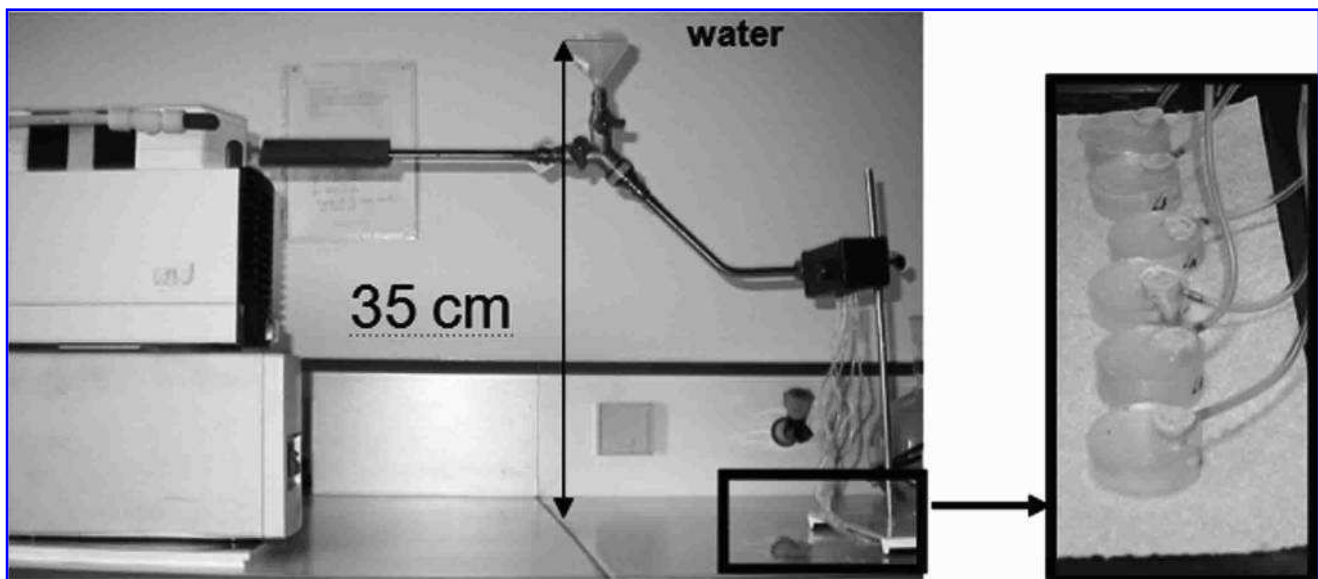


FIG. 1. Photograph of the pulpal pressure device used in this study.

TABLE 1. SHEAR BOND STRENGTH VALUES (MPa)

	<i>Clearfil SE Bond without pressure</i>		<i>Clearfil SE Bond with pressure</i>	
	<i>Bur</i>	<i>Er:YAG</i>	<i>Bur</i>	<i>Er:YAG</i>
Mean	13.87 <sup>a</sup>	9.62 <sup>b</sup>	7.99 <sup>b</sup>	9.52 <sup>b</sup>
Standard deviation	5.25	3.28	2.77	4.38

Different letters indicate statistically significant differences.

termine the types of failure present, which were classified as cohesive, adhesive, or mixed (both cohesive and adhesive failure zones present on the same fractured surface). These results have been detailed in reference 11 (in press).

Statistical analysis of the results was performed using the non-parametric Kruskal-Wallis test and the Mann-Whitney U test for independent samples, as well as Fisher's exact test. Statistical significance was set at  $p < 0.05$ .

In the second part of this study the SEM observations were performed in an environmental mode. (The SEM environmental node is dedicated to the observation of samples without metalization.) Eight dentine surfaces were divided into two groups of four teeth each, one group received pressure and the other group did not.

The surface of each tooth was divided into two parts: one was prepared with a bur and the other was prepared using the ER:YAG laser. The bonding protocol was the same except as that described above, except that the composite thickness was 1 mm and they were photopolymerized under glass pressure. Each specimen was divided into three longitudinal sections: two sections were sputter coated and examined with the SEM, and the last one was examined using the SEM in an environmental mode.

## Results

### Comparing shear bond strengths

Shearing experiments<sup>11</sup> were performed on the 80 teeth prepared as described above. Mean shear bond strength

measurements and standard deviations are presented in Table 1.

Without pressure, the shear bond strengths of the Er:YAG laser-prepared surfaces were lower than those seen on bur-prepared surfaces ( $p = 0.0053$ ). With pressure, the shear bond strengths of the bur-prepared surfaces and the laser-prepared surfaces were not statistically significantly different ( $p = 0.3867$ ). The shear bond strengths obtained for the bur-prepared surfaces were statistically significantly weaker with pressure than without ( $p = 0.0003$ ). The shear bond strengths obtained for the laser-prepared surfaces were nearly the same both with and without pressure ( $p = 1$ ).

### Comparing resin tags

Without pressure, long resin tags (around 10  $\mu\text{m}$ ) were seen on the surfaces prepared using the bur (Fig. 2A). On the surfaces prepared using the laser these tags were more numerous, longer, were bunched together (Fig. 2B).

With pressure, the resin tags were still numerous, but they were shorter (around 1  $\mu\text{m}$ ) on surfaces prepared using the bur (Fig. 3A). On surfaces prepared using the laser, we observed fewer resin tags and their diameters are smaller (Fig. 3B).

## Discussion

With the use of the intradentinal pressure device, the amount of moisture in the substrate was maximal. Actual clinical results would likely fall between the two sets of results obtained with and without pressure, since *in vivo* plasma proteins aggregate with the primer.<sup>12</sup> This aggregation causes a decrease in the diameters of the tubules, and thus a decrease in the amount of surface humidity present.

### Shear bond strength

As early as 1991, Mitchen and Gronas proposed that pressure decreases adhesive bonding strength, particularly in the absence of a smear layer.<sup>13</sup> Our results show that the smear layer does not appear to affect bonding strength. Indeed, the bonding values obtained with pressure on surfaces prepared with the bur (in the presence of a smear layer) and with the

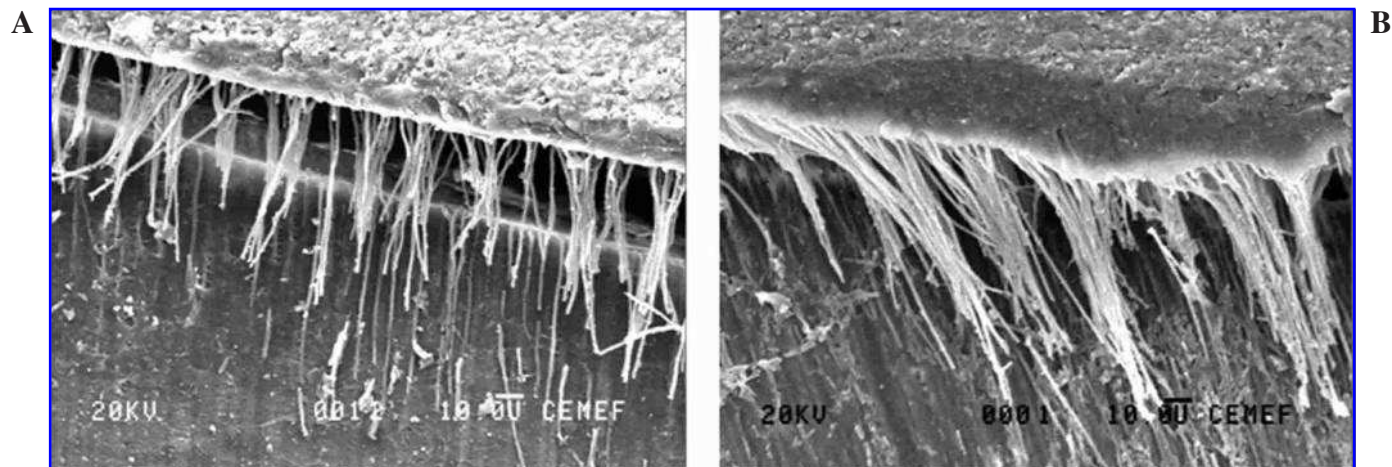


FIG. 2. Resin tags on surfaces prepared with the bur (A) or the laser (B) without intradentinal pressure.



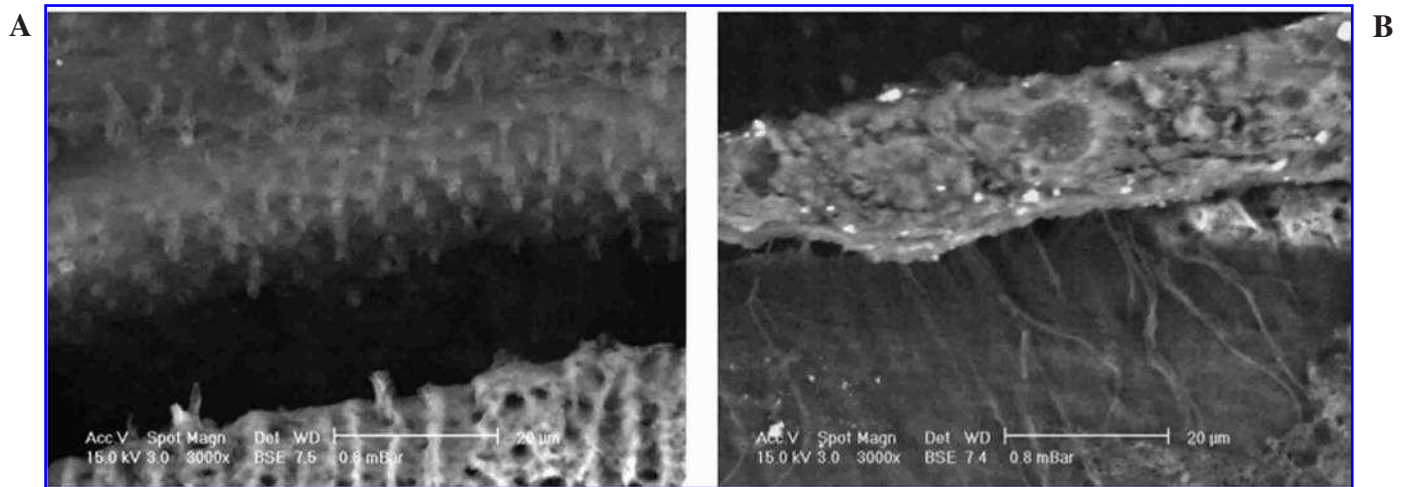


FIG. 3. Resin tags on surfaces prepared with the bur (A) or the laser (B) with intradental pressure.

laser (without a smear layer) were nearly the same. When we used the self-etching adhesive system on bur-prepared surfaces, the bond values decreased when intradental pressure was used during bonding.

The results we obtained from surfaces prepared with the bur along with intradental pressure are in good agreement with other studies in the literature. Most adhesives have lower bonding strength when used with pulpal or intradental pressure. In the work done by Nystrom et al. in 1998 using Scotch Bond MP, they found that even if the primer was hydrophilic, it did not penetrate into the demineralized dentine.<sup>14</sup> In 2000, Zeng et al. tested the Xeno III and the Dyract AP systems. They concluded that when under pressure, the primer does not penetrate properly because the HEMA was diluted before it was completely polymerized.<sup>15</sup> Pioch and Staehle tested the Syntac, Prime and Bond, and Gluma bonding systems. Their hypothesis was that the pressure would prevent the adhesive solvent from volatilizing.<sup>5</sup> Finally, Sengun et al. in 2003 tested the CLB2V system, and its composition is similar to that of the system we tested, Clearfil SE. The waiting periods before polymerization are slightly different for the two systems (30 sec for CLB2V and 20 sec for Clearfil SE). They also postulated that dilution of the HEMA was the reason for the decrease in bond strength seen when applied under pressure.<sup>16</sup>

#### Resin tags

Without pressure the Clearfil SE tags were more numerous on the surfaces prepared with the laser. One hypothesis for this is that the smear layer on the bur-prepared surfaces slowed the adhesive's penetration. Inversely, on the laser-prepared surfaces the tubules were wider, which made adhesive penetration easier.

With pressure, there were fewer tags with both types of surface preparation. One reason for this may be that the adhesive's hydrophilicity and its ability to penetrate tubules plays an important role when bonding to a dentine surface under intrapulpal pressure.

We found resin tags on bur-prepared surfaces both with and without intrapulpal pressure; however, the tags were extremely short when bonded under pressure. This may be

one of the reasons why bond values are lower for bur-prepared surfaces under pressure.

Concerning surfaces prepared using the laser, the absence of a smear layer enables the adhesive to bond to a cleaner surface. Thus the resin tags in those samples did not affect bond strength.

#### Conclusion

Here we compared the shear bond strength of a self-etching adhesive (Clearfil SE Bond) to surfaces prepared using an Er:YAG laser or a bur. Intrapulpal pressure was simulated to analyze its influence on shear bond strength. Shear bond strength values are clearly lower for bur-prepared surfaces when under intrapulpal pressure. Inversely, intrapulpal pressure did not significantly affect shear bond strength to laser-prepared surfaces. Intradental pressure also reduced the numbers of resin tags seen, but this did not significantly affect bond strength values to dentine prepared using the laser. The resin tags and the smear layer may play a role in the differences we found in our testing.

#### Disclosure Statement

No conflicting financial interests exist.

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